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# *Indian Standard*

# METHODS FOR MEASUREMENT OF AIR POLLUTION

## PART XVI RECOMMENDED PRACTICE FOR COLLECTION BY FILTRATION AND DETERMINATION OF MASS, NUMBER AND OPTICAL SIZING OF ATMOSPHERIC PARTICULATES

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# *Indian Standard*

# METHODS FOR MEASUREMENT OF AIR POLLUTION

## PART XVI RECOMMENDED PRACTICE FOR COLLECTION BY FILTRATION AND DETERMINATION OF MASS, NUMBER AND OPTICAL SIZING OF ATMOSPHERIC PARTICULATES

## 0. FOREWORD

**0.1** This Indian Standard ( Part XVI ) was adopted by the Indian Standards Institution on 31 January 1980, after the draft finalized by the Air Pollution Sectional Committee had been approved by the Chemical Division Council.

**0.2** Situations may arise where determination of mass and number, and optical sizing of atmospheric particulates may be necessary to meet diverse objectives principally connected with air pollution. In order to arrive at correct results, it is necessary to follow a standard, systematic method of determination.

**0.3** This standard is based on ASTM D 2009-65 'Standard recommended practice for collection by filtration and determination of mass, number, and optical sizing of atmospheric particulates' published by the American Society for Testing and Materials.

**0.4** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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## 1. SCOPE

**1.1** This standard ( Part XVI ) covers method for sample collection of particulate matter from an atmosphere by filtration and measurement of mass, amount, particle size, and particle size distribution of the collected material. Variations in the recommended practice permit sampling to meet a number of widely different assay needs. Although especially applicable to collection of solid particles, the filter method may be used also to collect liquid particles if determination of droplet size is not required.

*Rules for rounding off numerical values ( *revised* ).

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, definitions given in IS : 4167-1966* shall apply.

## 3. OUTLINE OF THE METHOD

**3.1** A measured and representative sample of the atmosphere under investigation is drawn through a filter medium selected to arrest and permit measurement of the particles that are to be studied. For most purposes, the exposed filter medium is removed from the sampling device and appropriate examination methods then applied to the collected matter. This recommended practice provides for determination of mass loading, particle concentration, particle size and size distribution and particle inspection. Chemical composition, radioactivity, and other characteristics of the particles may be determined by additional methods not included in this standard. Studies by phase microscopy, electron microscopy or other techniques also may be made on the collected material.

## 4. APPARATUS

**4.1 Sample Collector** — A typical arrangement of apparatus parts in a filter sampling system is given, schematically, in Fig. 1. These include a sampling nozzle, filter holder, filter medium, flow-measuring device, flow-inducing device, and means for regulating the flow of the sample air or gas.

**4.1.1** It is important that the filter be upstream from the rest of the apparatus so that any dirt in the system, manometer liquid or pump oil is not carried accidentally into the filter. The filter should be as close as possible to the sampling point, and any sampling lines should be perfectly clean. When sampling from a moving stream, as in a duct or stack, careful attention should be given to the shape and position of the nozzle. Recognized principles of stream sampling should be used otherwise the particles collected may not be representative of those in the test atmosphere. For sampling in the open, a nozzle is not needed.

**4.1.2** *Filter Holder* — The filter holder should be such as to hold the proper size of filter without waste of filtering area and without chance of channelling or leak around the filter. An inert metal screen or other mechanical support usually is required to prevent rupture or displacement of the filter in service. If the holder is not properly designed, flow through the medium will not be uniform over the face of the filter.

*Glossary of terms relating to air pollution.

Uniform flow distribution is especially desirable where particle counts are to be made. Many kinds of filter medium are available, and the one selected should best suit the purpose for which the particle collection is made.

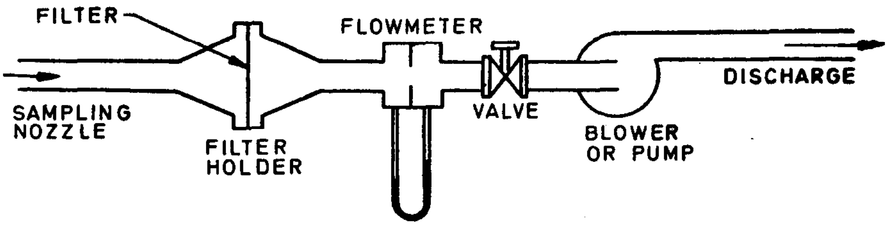


FIG. 1 TYPICAL ARRANGEMENT OF APPARATUS PARTS FOR PARTICLE COLLECTION BY FILTRATION

**4.1.3** *Flow Meter* — The flow meter may be of the orifice type, a rotameter, bellows gas meter, rotary gas meter, or other recognized device. In any case, it should be carefully calibrated under the conditions of use to give an accurate measure of sample volume or flow rate.

**4.1.4** Various types of air-moving equipment may be employed to draw the sample of air or gas through the system, the most usual being a turbine type blower, mechanical vane pump, piston or diaphragm pump, or ejector. For large volumes and for long sampling periods, turbine blowers are most satisfactory; multi-stage units are preferred since they will provide the necessary pressure drop. Mechanical pumps are particularly useful where there is high flow resistance in the system ( caused by special filters, long sampling lines, etc ). Except for flow rates of 0·028 $m^3$/min or less, ejectors are not recommended. If the system under test is at a higher pressure than some available exhaust point, or if there is a pressure drop across two parts of the system, it may be possible to use this existing pressure differential to draw the sample.

**4.1.5** Control of the sampling rate may be maintained by adjustment of a flow valve between the flowmeter and pump, by adjustment of a bleed valve at the pump inlet, or by use of a constant-flow orifice. It is sometimes possible and desirable to vary the voltage input into a mechanical pump motor in order to provide flow adjustment.

**4.2 Installed Collectors** — In areas where the atmosphere or other gas is to be sampled periodically or continuously, fixed sampling stations are often installed. These may be manual or automatic.

**4.3 Miniature Sampler** — A number of sample collecting units have been designed in miniature so that they are easily portable and capable of operation from an automobile or other battery power supply. Although the reduced size imposes some limitations ( for example, in sampling rate ), these units are acceptable if they conform to requirements of **4.1**.

**4.4 'High Volume' Sampler** — These are compact and portable collectors consisting of a large area filter holder, high-capacity blower, and built-in flow indicator. They may be designed to handle large flow rates of air up to 2·8 $m^3$/min or more.

**4.5 Membrane Filter Sampler** — This type of filter medium has a limited collection capacity but is ideally suited for particle counts, particle size measurement, study of particle characteristics and assay of living organisms.

**4.6 General Purpose Collectors** — Various designs and sizes of shop or laboratory-built collectors are possible, all of which may be applicable to this standard provided that the general considerations given in **4 1** are not violated.

**4.7 Laboratory Equipment for Particle Analysis**

**4.7.1** *Drying Oven* — Any reliable electric oven that may be operated continuously at a controlled temperature level of 110°C.

**4.7.2** *Weighing Balance* — A laboratory analytical balance capable of weighing to $\pm$0·05 mg.

**4.7.3** *Microscope* — General purpose type with magnification range to about 1 000 ×; eyepiece scale, whipple disk, or other reticule; stage micrometer; and suitable light source.

**4.7.4** *Miscellaneous Apparatus* — Lighweight metal cans with covers, desiccators and forceps.

**5. PRECAUTIONS**

**5.1** Many pumps tend to give off a fine fog of lubricant that can quickly contaminate a filter. This contamination should be avoided either by placement of an efficient clean up filter on the pump discharge line to remove the oil fog, or by ensuring that the discharge is vented out of doors or well downwind of the filter.

**5.2** Automobile exhausts are another source of oil fog that should be avoided. When sampling over long periods of time out of doors, protection should be provided against rain. Most filters are seriously affected when they become wet.

**5.3** During the sampling period the filter tends to become filled and the flow rate may decrese as filter resistance increases. Therefore, it may be necessary to adjust the flow rate frequently or provide for essentially constant flow automatically ( as with a critical flow orifice ). Otherwise an accurate record of flow rate throughout the sampling period should be maintained.

## 6. SAMPLING

**6.1 Sampling** — Sampling should be carried out as prescribed in IS : 5182 ( Part XIV )-1979*.

**6.2 Selection of Sampler and Filer Media** — Based upon the purpose for which the sample is to be taken, determine from Table 1 the sampler to be used and the appropriate filter medium ( *see* Note 1 ). Follow that method below which is applicable. Where very small particles ( 1·0 µm or less in diameter ) are of special interest, a high-efficiency filter medium should be used.

NOTE 1 — There are many types of filter media available and any one may be used, provided that it has the proper performance characteristics. The four indicated in Table 1 have been selected as capable of meeting most situations that may be encountered.

NOTE 2 — Samples for size measurements may be collected with advantage by inertial samplers like casella cascade impactor. The flow rate for this type of sampler is 17·5 l/min. The procedure to be followed is as follows:

Load the first glass stage in the impactor and sample for 23 minutes. Load the second stage after cleaning the support and sample for next 5 minutes. Load the third stage and sample for one and a half minutes. The inspector should be brought to the laboratory and unload at the time of counting. This method has an advantage over the filter paper method as the crowding of small particles in the sample is avoided. For collection of samples on microscope slides for their size estimation a thermal precipitator may also be used. Glass slides from the individual stages of the cascade impactor and microscope slides from the thermal precipitator may be examined in accordance with the method given in **7.2.4** and **7.2.4.1**.

## 7. PROCEDURE

**7.1 Mass Concentration** — Use either the high volume sampler or a general purpose, laboratory-built unit, as described in **4.6.** For total suspended matter, determine the initial mass of the filter by weighing it accurately on the analytical balance. The filter should be conditioned before each weighing so that the moisture content does not interfere with the determination mark for identification and weigh a lightweight metal can with its cover. Place the filter in the can and leave the can open in an oven at 110°C for 1 hour. If the collected material is temperature-sensitive, use a desiccator in place of oven. Cover the can and allow it

*Method for measurement of air pollution : Part XIV Guidelines for planning the sampling of atmosphere.

to cool, preferably in the desiccator. Weigh the closed can with its contained filter. Expose again in the oven for 1 hour and repeat the cooling and weighing. Continue until a constant mass is obtained. Glass fibre media have very little moisture absorption, and therefore, show excellent mass stability and are particularly useful for low-temperature drying in a desiccator.

**TABLE 1 SAMPLING EQUIPMENT AND MEDIA APPLICABLE TO THE FILTER METHOD FOR DETERMINATION OF PARTICULATE MATTER IN THE ATMOSPHERE**

| Purpose of Sample | Applicable Media | | | |
|---|---|---|---|---|
| | Acid-Washed Cellulose Filter Paper | High-Efficiency Glass Fibre Paper | High Velocity | Membrane Medium |
| Mass concentration | 1, 2, 3 | 1, 2, 3 | 2 | 1, 3, 4 |
| Chemical analysis | 1, 2, 3 | 1, 2, 3 | 2 | 1, 3, 4 |
| Particle count | ... | ... | ... | 1, 3, 4 |
| Particle sizes | ... | ... | ... | 1, 3, 4 |
| Particle inspection | ... | ... | ... | 1, 3, 4 |

The applicable sample collectors are as follows:

1 — General purpose sampler,

2 — High-volume sampler,

3 — Miniature sampler, and

4 — Membrane filter apparatus.

**7.1.1** Place the filter in the sampler and operate for the required length of time depending on the purpose of the test. Remove the filter from the sampler carefully so that none of the collected matter or any of the filter structure is lost. Replace in the same weighing can previously used for that filter and recondition in the same manner for the time period previously found. Cover the can, cool, and weigh. Expose the can open for 1 additional hour, close, cool ( if necessary ), and weigh again to make sure that the mass is constant. Subtract from this mass the original mass of the can, cover, and filter, and record the difference as the mass of collected matter.

**7.2 Particle Size and Size Distribution** — Use either the commercially available samplers or the general purpose unit as described in **4.6.** Membrane filters are best for this determination.

**7.2.1** Remove the filter from the sampler carefully so that none of the collected matter is lost. Use forceps in handling the filter. If it is not possible to examine the sample immediately after collection, store the filter in a closed container such as a small petri dish.

**7.2.2** For ease in handling during analysis, circular filters may be cut into 4 segments of equal area or the filter may be examined in its entirety if a microscope slide of adequate size is available. Hold the edge of the filter with a forceps and roll a glass rod that has been wet with immersion oil ( index of refraction about 1·51 ) along the back or underside of the filter. The immersion oil will be absorbed and the filter will become transparent. Place the wet filter on a clean microscope slide with collection surface upward. Cover the collection surface with a cover glass of No. 1 thickness.

**7.2.3** The magnification employed for examining the collection depends upon the diameter of the particles. A useful magnification is given by the relation:

$$\text{Magnification} = 700/d$$

Where $d$ is the average diameter of the particles in micrometre. For magnifications above 500 ×, use oil immersion and a suitable objective lens.

**7.2.4** If a range of sizes is present, several magnifications may by used. Particle sizes down to 0·4 $\mu$m in diameter may be sized with the optical microscope. It is possible to count smaller particles but is not possible to resolve to permit size estimation. Evaluate the size range using low power ( 20 × to 40 × ) to look for large particles and high power ( up to approximately 1 000 × ) to look for small particles. If the particles are all of one size, the analysis is greatly simplified. If there is a size range, the range may be broken down into groups. The range is arbitrary and may be selected to fit the particle image to a scale superimposed on the field of view as in a filar micrometer or by a reticule located in the microscope eyepiece. In either case, calibrate the eyepiece scale against the engraved lines on a stage micrometer. Count particles larger than 20 $\mu$m in diameter at low magnification ( approximately 140 × ). During this step, ignore particles smaller than 20 $\mu$m. Count and record the number of particles in each microscope field in each of the larger subgroups. The field is usually arbitrarily defined as the total area included within the working portion of the reticule. Continue this counting of each subgroup in randomly selected fields until enough counts have been made to assure adequate reliability. In general, reliability improves with the number of counts. As a guide, the product of the total number of particles in each subgroup and the number of fields counted may be taken as 1 000. In some cases, the number of particles

may be so low that it is not possible to attain the product 1 000. In this case, continue counting until 1 percent of the total filter area has been counted. The number of fields to be counted is illustrated as follows:

| *Total Number of Particles Observed in a Size Range* | *Number of Fields to be Counted* |
|---|---|
| 250 | 4 |
| 100 | 10 |
| 50 | 20 |
| 10 | 100 |
| 1 | 1 000 |

**7.2.4.1** For the next smaller range of particles, between 2 and 20 µm, for example, use a higher magnification ( approximately 400 × ). At a higher magnification make no counts below the minimum size selected nor above the maximum. Count and size particles within this size range. For the smallest particles, magnifications up to 1 000 × or higher may be desirable. At this highest magnification record the number and size of visible particles smaller than the previously selected minimum. From the total number of particles observed in each size range, the area scanned, the sample volume, and the total filter area, calculate particles size and size distribution.

## 8. REPORT

**8.1** The results may be presented in any convenient form. Usually, the results are presented graphically.

( *Continued from page* 2 )

## Methods of Sampling and Analysis Subcommittee, CDC 53 : 2

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**RELATING TO AIR POLLUTION**

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4167-1966 Glossary of terms relating to air pollution

5182 Methods for measurement of air pollution:

- ( Part I )-1969 Dustfall
- ( Part II )-1969 Sulphur dioxide
- ( Part III )-1970 Radioactivity ( particulate ) in air
- ( Part IV )-1973 Suspended matter
- ( Part V )-1975 Sampling of gaseous pollutants
- ( Part VI )-1975 Nitrogen oxides
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- ( Part XII )-1974 Polynculear aromatic hydrocarbons in air particulate matter
- ( Part XIV )-1979 Guidelines for planning the sampling of atmosphere
- ( Part XV )-1974 Mass concentration of particulate matter in the atmosphere
- ( Part XVI )-1980 Recommended practice for collection by filteration and determination of mass, number and optical sizing of atmospheric particulates
- ( Part XVIII )-1974 Continuous analysis and automatic recording of the oxidant content of the atmosphere

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9005-1978 Limits for gaseous emissions from nitric acid and nitrogenous fertilizer industries

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